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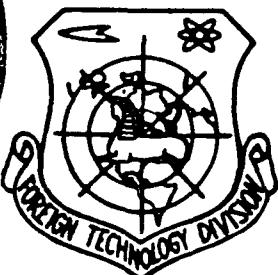


PROTECTIVE COATINGS FOR NIOBIUM ALLOYS AND THEIR PROPERTIES

by

Ye. L. Geraseva, A.N. Sokolov, et al.

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Block	Italic	Transliteration .	Block	Italic	Transliteration
А а	А а	A, a	Р р	Р р	R, r
Б б	Б б	B, b	С с	С с	S, s
В в	В в	V, v	Т т	Т т	T, t
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*ye initially, after vowels, and after t, b; e elsewhere.
When written as е in Russian, transliterate as ye or е.

RUSSIAN AND ENGLISH TRIGONOMETRIC FUNCTIONS

Russian	English	Russian	English	Russian	English
sin	sin	sh	sinh	arc sh	\sinh^{-1}
cos	cos	ch	cosh	arc ch	\cosh^{-1}
tg	tan	th	tanh	arc th	\tanh^{-1}
ctg	cot	cth	coth	arc cth	\coth^{-1}
sec	sec	sch	sech	arc sch	sech^{-1}
cosec	csc	csch	csch	arc csch	csch^{-1}

Russian	English
rot	curl
lg	log

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PROTECTIVE COATINGS FOR NIOBIUM ALLOYS AND THEIR PROPERTIES*

Ye. L. Geraseva, A. N. Sokolov, A. S. Stroyev, N. F. Lashko,
M. N. Kozlova, and I. I. Titarenko

Niobium alloys cannot be used for operation at high temperatures without /139 protective coatings. Superficial alloying of niobium alloys with only one element (aluminum, silicon or chromium) does not make it possible to obtain a coating which reliably protects the alloy for a prolonged period of time. The development of complex coatings is still proceeding only by empirical means, since the chemism of their formation reactions is unknown. The study of the chemical and phasal compositions of complex protective coatings has great significance, since this allows us to judge through the end products of high-temperature reactions about the products themselves.

This work studied chromium-aluminum-silicon and chromium-titanium-silicon coatings which were applied to alloys VN-2A and VN-4 in two stages. First the alloys were subjected to saturation with chromium, aluminum, or chromium and titanium, and then siliconing was performed. The chemical and phasal composition of the coating were determined by the method of by-layer phasal analysis and the method of microprobe. A metallographic analysis was also performed, heat-resistance curves were plotted, and certain mechanical properties were determined.

The by-layer phasal analysis was performed by means of electrochemical dissolution of the surface layers of a certain thickness with subsequent chemical analysis of the anode sediment and electrolyte. Dissolution of the alloy with the coating was conducted in an electrolyte consisting of 1% hydrochloric acid ($d=1.19$) in absolute ethyl alcohol with current density of $0.03-0.04 \text{ a/cm}^2$. In a number of cases the surface layer did not conduct electrical current. Then the electrolytic method of dissolution was combined with the chemical. We performed the dissolution of the non-conducting layer in hydrofluoric acid with addition of nitric acid.

The study of the chemical and phasal composition was conducted also /142

*I. V. Kuznetsova and V. F. Shestova also participated in the work.

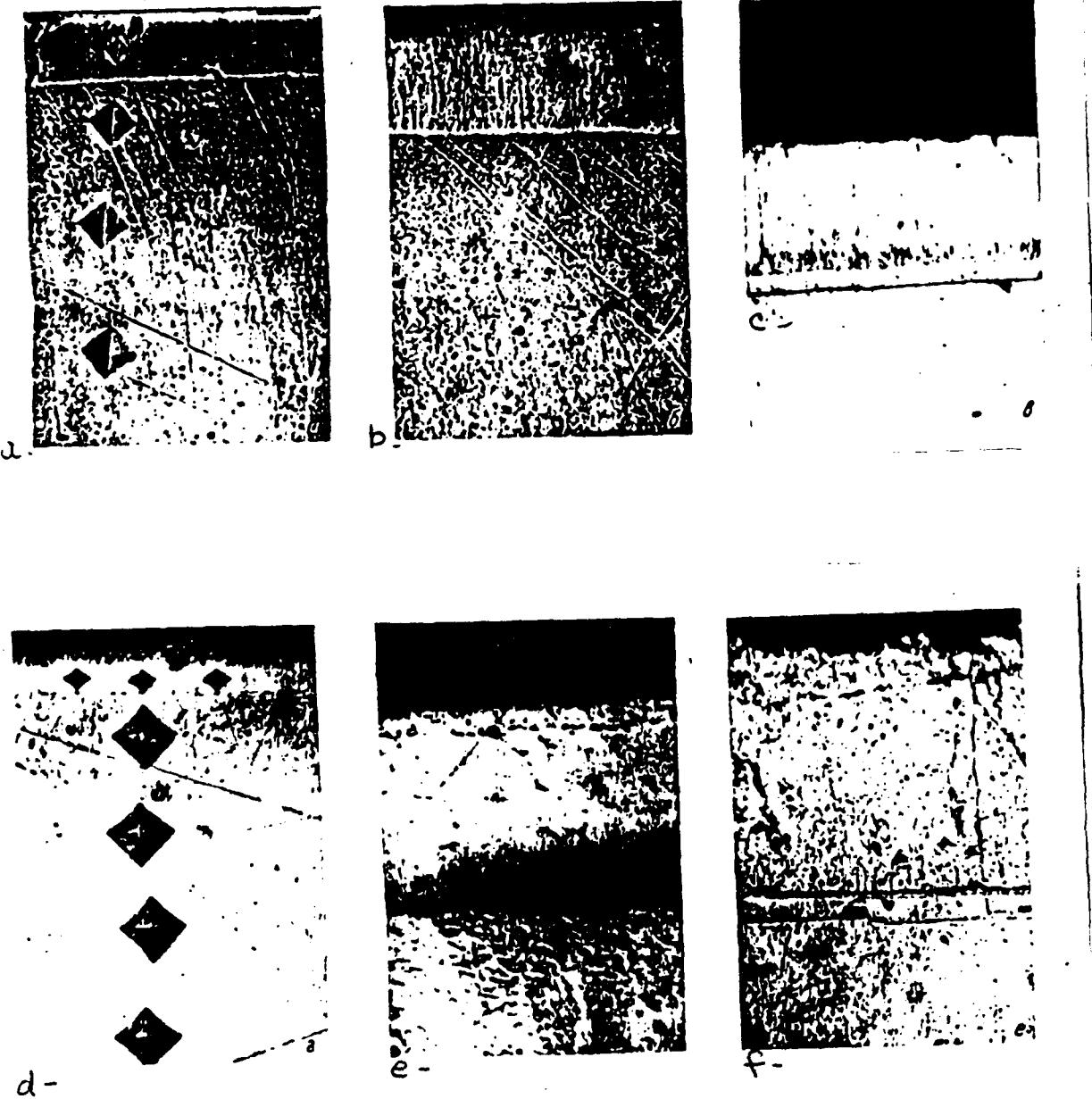


Fig. 1. Microstructures of surface layer of alloys VN-2A and VN-4. Magnified x450
 a - alloy VN-2A, chromalitized; b - alloy VN-2A, chromalitized with subsequent siliconing; c - alloy VN-2A, coating Cr-Al-Si + heating at 1100°C, 100 hrs;
 d - alloy VN-4, chromatitanized; e - alloy VN-4, chromatitanized with subsequent siliconing; f - alloy VN-4, coating Cr-Ti-Si + heating at 1100°C, 100 hrs.

by the microsound method on a MAR-1 device with accelerating voltage of 35 kV. The locality of the method was around 3 m. Mica and quartz (1340) served as the crystal-analyzers. The distribution of component elements in the surface layer was studied on diagonal and straight sections. The obtained curves were re-computed into percentages with consideration for the correction for absorption.

The structure of the surface layer was determined by the URS-50IM x-ray diffractometer. Photography was performed on copper irradiation with the application of a standard (silver). The rate of oxidation was determined by added weight by the method of weighing with periodic cooling. The samples were tested for heat resistance according to regimen 20-700-20, 200-1100-200°C (heating for 30 seconds, holding for 2 minutes, cooling for 30 seconds). The plastic properties were determined by the bend angle of the samples heated to temperatures of 700 and 1100°C.

Chromium-aluminum-silicon coating. The microstructure of the surface layer of the sample of alloy VN-2A after saturation with chromium and aluminum is shown in Fig. 1,a. The phasal by-layer analysis of the chromium-aluminum coating is performed in Table 1. As the x-ray analysis showed, the diffusion layer represents an intermetallide phase Cr_2Nb of the structural type Cu_2Mg .

The diffusion layer after siliconization^{of} the chromalitized sample is shown in Fig. 1,b. As we can see from the data of the phasal analysis (Table 2), it consists of particles of niobium disilicide of varying dispersity, niobium carbide, and a solid silicon solution in niobium. The intermetallide phase Cr_2Nb was not found.

Table 1. Phasal by-layer analysis of chromium-aluminum coating on alloy VN-2A

a - Расстояние за поверхно- стн, мкм	b - Убыль веса покрытия, г	c - Содержание элементов в % к убыли веса покрытия, сплава			
		d - в анодном осадке		e - в электролите	
		Cr	Nb	Cr	Nb
5	0.0330	0.30	f - следы	15.96	68.62
9	0.0272	0.19	*	3.84	-
15	0.0374	0.13	*	1.96	88.58
17	0.0716	0.08	*	2.16	92.15

a - Distance on surface, μm ; b - Loss of coating weight, g; c - Content of elements in % of loss of coating weight, alloy, d - in anode precipitate; e - in electrolyte; f - traces.

Table 2. Phasal by-layer analysis of alloy VN-2A with chromium-aluminum-silicone coating

a - Рассто- жение от поверхно- сти, мкм	b - Убыль веса покрытия, г	c - Содержание дисилицида ниобия, ниобия, молибдена и кремния в % к убытку веса покрытия, сплава						
		d - в анодном осадке			e - в электролите			
		NbSi ₂	Nb	Mo	Nb	Mo	Si	
5	0.0402	1.99	f - Нет	g - Следы	51.11	2.80	36.84	
10	0.0412	1.46	•	0.01	53.30	3.09	35.83	
16	0.0460	0.65	•	0.04	60.50	3.44	21.02	
22	0.0540	7.03	•	0.03	—	—	13.15	
31	0.0704	16.05	0.78	0.07	71.42	3.25	7.63	
40	0.0716	12.01	0.65	0.05	77.69	3.61	4.24	
47	0.0640	4.06	1.03	0.07	—	—	—	
53	0.0611	3.21	0.60	0.07	83.34	3.90	3.30	
60	0.0632	3	—	—	—	—	—	
67	0.0652	3	Следы	1.15	0.10	86.16	3.90	1.28

a - Distance from surface, μm ; b - Loss in weight of coating, g; c - Content of niobium disilicide, niobium, molybdenum and silicon in % of loss of weight of coating, alloy; d - in anode precipitate; e - in electrolyte; f - None; g - Traces.

After ordinary silicone coating, the surface layer contains 3-7% coarsely disperse particles of niobium disilicide [1]. After silicone coating of the chromalitized samples, the surface layer of a certain depth contains a small amount of coarsely disperse particles of niobium disilicide (0.65-2%), and in deeper layers 7-16%. The surface layer is enriched with chromium.

Upon heating the sample with the chromium-aluminum-silicon coating, the niobium disilicide oxidizes, with formation of a glass-like layer of silica (silicon dioxide), which is insoluble by the electrochemical method. The size of the silica layer increases with the increase in temperature /144 and duration of heating. Heating the sample causes diffusion of the silicon into the base metal. On the boundary with the base metal, an interlayer of phase Nb_5Si_3 appears (Fig. 1,c).

Table 3. Phasal by-layer analysis of sample with chromium-aluminum-silicon coating after heating at a temperature of 1100°C for 100 hours.

a - рассто- яние от поверх- ности, мкм	b - убыль веса по- крытия, сплава, г	c - Содержание дисилицида ниобия, ниобия, молибдена хрома, кремния в %, и убыли веса покрытия, сплава								
		d - в анодном осадке				e - в электролите				
		NbSi,	Nb	Mo	Cr	Nb	Mo	Cr	Si	
5	0.0205	f - —	f - Нет	0.39	0.20	15.73	0.91	0.22	—	
15	0.0526	f - Нет	1.14	0.06	0.02	65.94	2.45	0.24	20.42	
35	0.1081	3.70	—	—	—	—	—	—	—	
50	0.0724	7.88	3.35	0.07	0.03	69.49	2.55	0.05	3.72	
64	0.0690	5.22	—	—	—	—	—	—	—	
76	0.0606	Следы	1.36	0.07	0.02	80.96	2.96	0.02	1.62	

a - Distance from surface, m; b - Loss in weight of coating, alloy, g;
 c - Content of niobium disilicide, niobium, molybdenum, chromium, and silicon in % of loss of weight of coating, alloy; d - in anode precipitate; e - in electrolyte; f - None; g - Traces.

The phasal composition of the sample with chromium-aluminum-silicon coating after 100-hour heating at 1100°C in air is presented in Table 3.

The chromium-aluminum-silicon coating is more stable in terms of its heat resistance than the ordinary silicon coating. The heat resistance of such a coating is fully satisfactory. The coating withstood over 500 cycles of heating and cooling without disintegration.

The long-term durability of the alloy with the coating is somewhat lower than without the coating (Table 4,5). The protective layer is retained after disintegration of the sample during testing for long-term strength.

Table 4. Mechanical properties of niobium alloys with and without coatings

a - Состояние материала	b - испыт., °C	c _s , кГ/мм ² , kg/mm ²	c ₀ , кГ/мм ² , kg/mm ²	e	δ, %	φ, %
c - Без покрытия	20	70.0	53.5	—	18.4	46.5
" "	20	67.0	53.0	—	22.0	61.5
Cr-Al-Si	20	73.5	55.5	—	10	10.3
d - То же	20	72.5	56.0	—	9.6	10.0
" "	20	67.3	—	—	13	24
Cr-Ti-Si	20	50	36	34	22	63
d - То же	20	44	34	30	24	63
" "	700	40	32	29	15	—
" "	700	41	33	30	13	68
Cr-Al-Si	900	47.5	43.5	—	—	17.5
d - То же	1050	38	36.5	—	—	33.5

a - State of material; test temp, °C; c - Without coating; d - Same.

Table 5. 100-hour long-term strength of niobium alloy with and without coatings.

a - Состояние материала	b - тест., °C	c - Механические свойства		
		$\sigma_{\text{рн}}$, кГ/мм ²	δ , %	ψ , %
d - Без покрытия	700	38	31	31
e - Cr-Ti-Si-покрытие	700	31	—	—
f - Cr-Al-Si-покрытие	900	33	—	—
g - То же	1050	20	—	—
g - ,	1100	14	14	19
e - Cr-Ti-Si-покрытие	1100	14	20	63
g - То же	1100	14	—	—
g - ,	1100	15	1	—
d - Без покрытия	1100	16	—	—

a - Condition of material; b - test temperature, °C; c - Mechanical properties;
d - Without coating; e - Cr-Ti-Si coating; f - Cr-Al-Si coating; g - Same.

Table 6. Coefficients of linear expansion of chromium-aluminum-silicon coating.

a - T° обр., град.	$d, \text{ \AA}$	$\alpha \cdot 10^{-4}$ град. ⁻¹ (deg)	$d, \text{ \AA}$	$\alpha \cdot 10^{-4}$ град. ⁻¹ (deg)	$d, \text{ \AA}$	$\alpha \cdot 10^{-4}$ град. ⁻¹ (deg)	$d, \text{ \AA}$	$\alpha \cdot 10^{-4}$ град. ⁻¹ (deg)	$d, \text{ \AA}$	$\alpha \cdot 10^{-4}$ град. ⁻¹ (deg)
20	0.7739	9.5	0.7763	9.5	0.7821	9.3	0.7911	9.5	0.8074	9.5
800	0.7796	8.4	0.7821	10.9	0.7877	8.4	0.7970	10.2	0.8134	10.3
920	0.7804	10.1	0.7831	10.7	0.7885	10.1	0.7980	10.1	0.8144	10.7
1020	0.7812	10.2	0.7840	10.8	0.7843	10.2	0.7988	10.8	0.8152	10.8
1200	0.7826	10.2	0.7855	10.4	0.7907	10.1	0.8003	10.2	0.8169	10.6
1300	0.7834	10.2	0.7863	10.4	0.7915	10.1	0.8011	10.2	0.8178	—

a - T° of sample, degrees.

The coefficients of heat expansion of the chromium-aluminum-silicon coating in a temperature interval of 20-1300°C were also determined. The determination was performed by the x-ray method on a heavy-duty sharp-focus tube with copper anode in a special high-temperature chamber. The heating of the sample was performed by the resistance method. The temperature was measured with the aid of an optic pyrometer OPIR-09.

The measurement of the linear expansion coefficients was performed along five lines (interplanar distances for these lines and the values of computed according to them are presented in Table 6).

The coefficient of linear expansion α was computed according to the formula

$$\alpha = \frac{\Delta d}{d_1 \Delta T}$$

where $\Delta d = d_2 - d_1$ is the change in interplanar distance during heating of the sample in a temperature interval of $T^o = T_2 - T_1$.

From Table 6 we see that the linear expansion coefficient of the protective layer of Cr-Al-Si on the alloy VN-2A changes very little with temperature. In the temperature interval of $20-800^oC$ the average value of the linear expansion coefficient is $\alpha = 9.5 \cdot 10^{-6}$, and in a temperature interval of $800-1300^oC$ the coefficient is $\alpha = 10.2 \cdot 10^{-6} \text{ deg}^{-1}$. The average value of the linear expansion coefficient in an interval of $20-1300^oC$ comprises $9.8 \cdot 10^{-6} \text{ deg}^{-1}$ (Fig. 2).

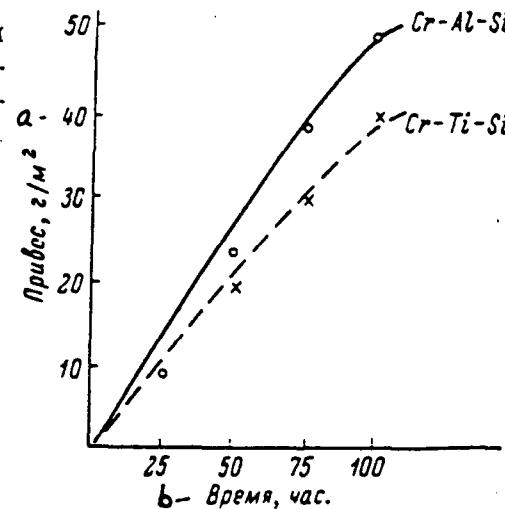


Fig. 2. Curves of heat resistance of alloys VN-2A and VN-4 with coatings at temperature of 1100^oC .

a - Added weight, g/m^2 ; b - Time, hours.

Chromium-titanium-silicon coating. After saturation of the alloy VN-4 comprised of chromium-titanium alloy, the diffusion layer consists of a phase $\text{Cr}_2(\text{Nb}, \text{Ti})$ of the type Cu_2Mg with parameter of elementary nucleus 6.948 \AA^0 . The ratio of chromium to the sum of niobium and titanium in the phase is 2 : 1 (the phase also contains around 2% molybdenum). Fig. 1,d shows the microstructure of the surface layer of alloy VN-4 after chromotitanization, while Fig. 3 shows the distribution of alloying elements in the chromotitanized layer, determined by the microsound method.

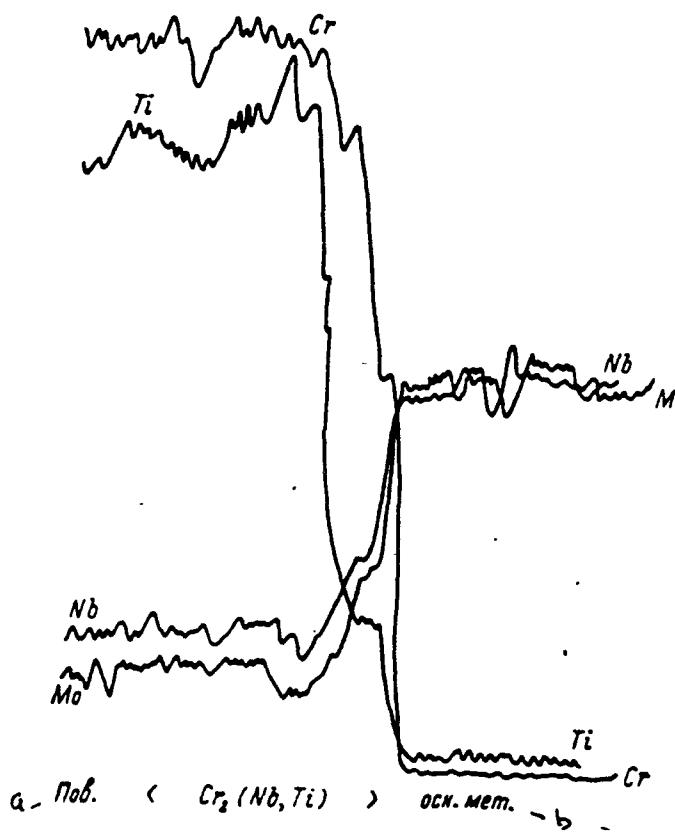


Fig. 3. Distribution of elements in the surface layer of alloy VN-4 after chromotitanization.

a - Coating; b - Base metal.

After siliconization^{of} the chromotitanized sample according to the data of the x-ray structural analysis, confirmed also by the results of the microsound (Fig. 1, b and 4), the protective coating consists of two layers. A thin layer on the surface represents silicide $NbSi_2$, enriched with chromium and titanium. The main portion of the coating consists of the phase $NbSi_2$ without chromium and titanium, but with a slight content of molybdenum. The phase $Cr_2(Nb, Ti)$ in the protective coating was not found. /147

Fig. 1,f shows the microstructure of the surface layer of alloy VN-4 with chromium-titanium-silicon coating after 100 hours of heating at $1100^{\circ}C$. The corresponding picture of the distribution of alloying elements in the surface layer is presented in Fig. 5. As we can see from Fig. 5, several oxides have formed on the surface: Cr_2O_3 , TiO_2 (rutile), SiO_2

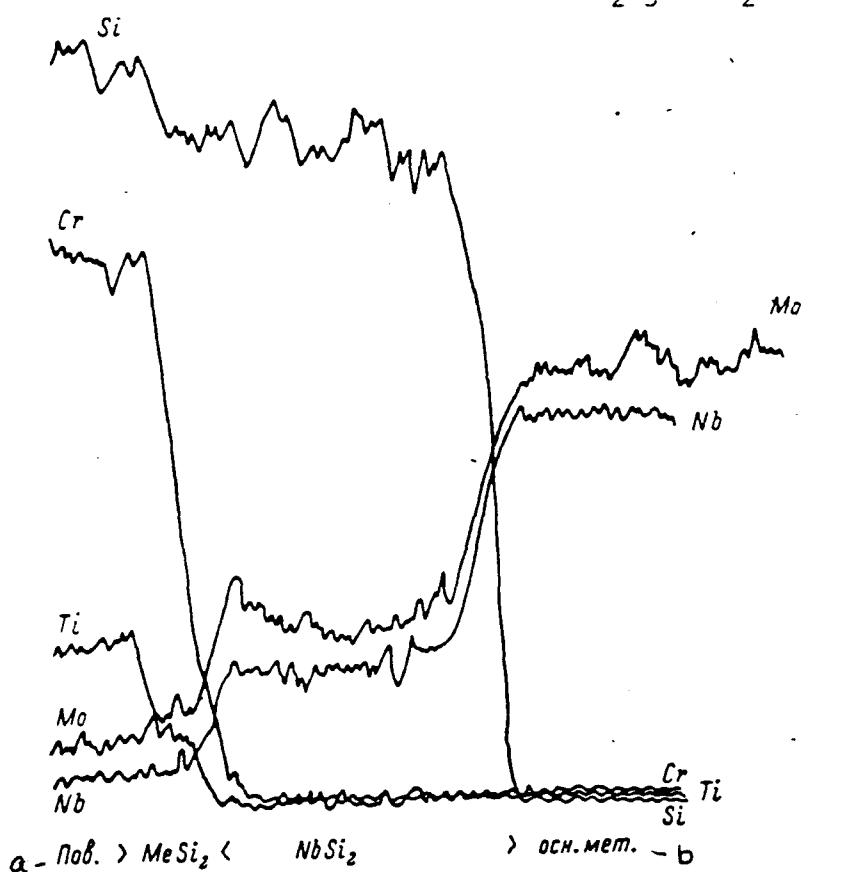


Fig. 4. Distribution of elements in the surface layer of alloy VN-4 after chromotitanization and subsequent siliconization.
a - Coating; b - base metal.

and $\beta\text{-Nb}_2\text{O}_5$.* The formation of several oxides of different crystalline structure must reduce the heat-resistant properties of the coating due to cracking of the oxide film. As demonstrated by metallographic study, the oxidation in this case is intensively dispersed along the boundaries of the coating grains to the base metal. Just as in the chromium-aluminum-silicon coating, a Nb_5Si_3 phase appears on the boundary with the base metal after prolonged heating.

The chromium-titanium-silicon coating has good heat resistance (Fig. 2). As tests demonstrated, the rate of oxidation in the initial hours is notably higher than in subsequent ones, and depends on the titanium content in the surface layer. Oxidation with testing time in excess of 50 hours is subject to parabolic law.

The chromium-titanium-silicon coating withstands no less than 500 cycles in testing for heat resistance. After testing for heat resistance, the samples were heated at 1100°C for 50 hours. No disintegration of the coating was noted.

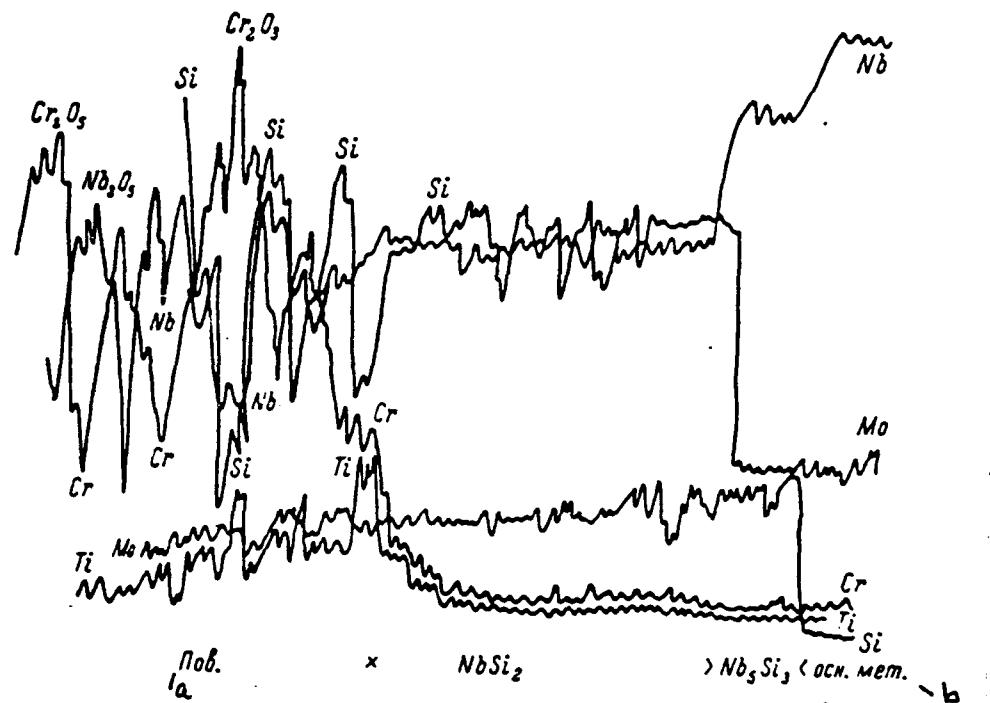


Fig. 5. Distribution of elements in the surface layer of alloy VN-4 with coating Cr-Ti-Si, heated at 1100°C for 100 hours.

a - Surface; b - base metal.

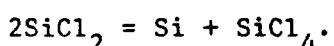
* Microsound data were confirmed by the results of x-ray structural study of the surface layer.

The bend angle for the alloy with the chromium-titanium-silicon coating is equal to 40 and 70° for temperatures of 700 and 1100°C. The mechanical properties of the alloy with the coating at room temperature is somewhat lower than without the coating. At high temperatures there is an even greater reduction in the mechanical properties (Tables 4, 5).

The microhardness by depth of the chromium-titanium-silicon coating is found within the limits of 1227-1287 kG/mm² and changes little after testing for heat resistance. The hardness is higher with higher content of titanium in the layer. The hardness of the base metal is equal to 185-225 kG/mm² and does not change after the test for heat resistance.

Discussion of results. In formation of chromium-aluminum-silicon and chromium-titanium-silicon coatings on niobium alloys, a certain general /149 regularity may be observed. After saturation with chromium-aluminum or chromium-titanium hardener, the surface layer consists of the phase Cr₂Nb [or Cr₂(Nb, Ti)]. Subsequent siliconization yields a diffuse layer whose main part is comprised of niobium disilicide NBSi₂. After siliconization, the Cr₂Nb phase is not found in both cases, either by the method of phase analysis or by the microsound method.

Upon saturation with a hardener containing an activator (ammonium chloride), the formation of diffusion layers takes place as a result of the reactive diffusion with participation of silicon atoms, which are formed on the surface of the saturated metal by the disproportionation reaction:



Active silicon (in statu nascendi) enters the reaction with the phase Cr₂ (Nb, Ti), of which the surface layer consists prior to silication. Since the silicon is in thermodynamic equilibrium with the phase of the structural type CrSi₂, then as a result of the interaction of silicon with niobium or silicon with chromium, it is specifically this phase which primarily emerges. A phase of the same structural type is obtained upon interaction of silicon with niobium, chromium and titanium bonded in compound Cr₂ (Nb, Ti). In this case, a solid solution of isomorphic disilicides of the type CrSi₂ is formed. The structural formula for the obtained compound should have the appearance of: [(Nb, Cr, Ti)Si₂]. New portions of disilicides are formed as a result of the predominant diffusion of silicon through

the formed layer of silicides and its interaction with the alloy (solid solution of molybdenum in niobium). The formed disilicide has the same structural type, but does not contain chromium and titanium.

The course of the process of saturation may be changed if we reduce or completely exclude chlorides from the saturating mixture, which corresponds to a reduction in the rate of occurrence of the disproportionation reaction.

Fig. 6. shows the distribution of elements in the surface layer of the sample ^{siliconized} in a mixture in which the amount of chlorides was reduced by two times as compared with the previous mixture. From Fig. 6 we may see that the main portion of the layer also consists os disilicide based on NbSi_2 . However, the thin surface layer differs from the previous one by its crystalline structure and by its ratio of elements. It represents the phase $[(\text{Cr}, \text{Ti}, \text{Nb})\text{Si}_2]$ with crystalline lattice of the ZrSi_2 type. With oxidation of the alloy VN-4 having such a coating, up to holding for 100 hours at 1100°C , only chromium oxides are formed on the surface. The properties of the coating become more stable, and oxidation along the grain boundaries of the coating occurs to a much lesser degree.

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Fig. 7 shows the distribution of elements in the surface layer of alloy VN-4 ^{siliconized} without chlorides. The layer consists of two zones. On the surface we find disilicide $(\text{Nb}, \text{Cr}, \text{Ti})\text{Si}_2$. Part of the layer of the intermetallide phase $\text{Cr}_2(\text{Nb}, \text{Ti})$ has been retained on the boundary with the base metal.

The resistance of the coating against oxidation significantly depends on the chemical and phasal composition of the first zone of the diffuse layer, since they determine the structure and composition of the oxides formed on the surface. The formation of various oxides leads to cracking of the oxide film, to development of oxidation along the boundaries of the grains in the coating, and to a reduction in the stability of the coating's properties.

The homogeneous oxide film which is formed, for example, during oxidation of a sample siliconized in a mixture with reduced amount of chlorides facilitates the stabilization of the mechanical characteristics of niobium alloys.

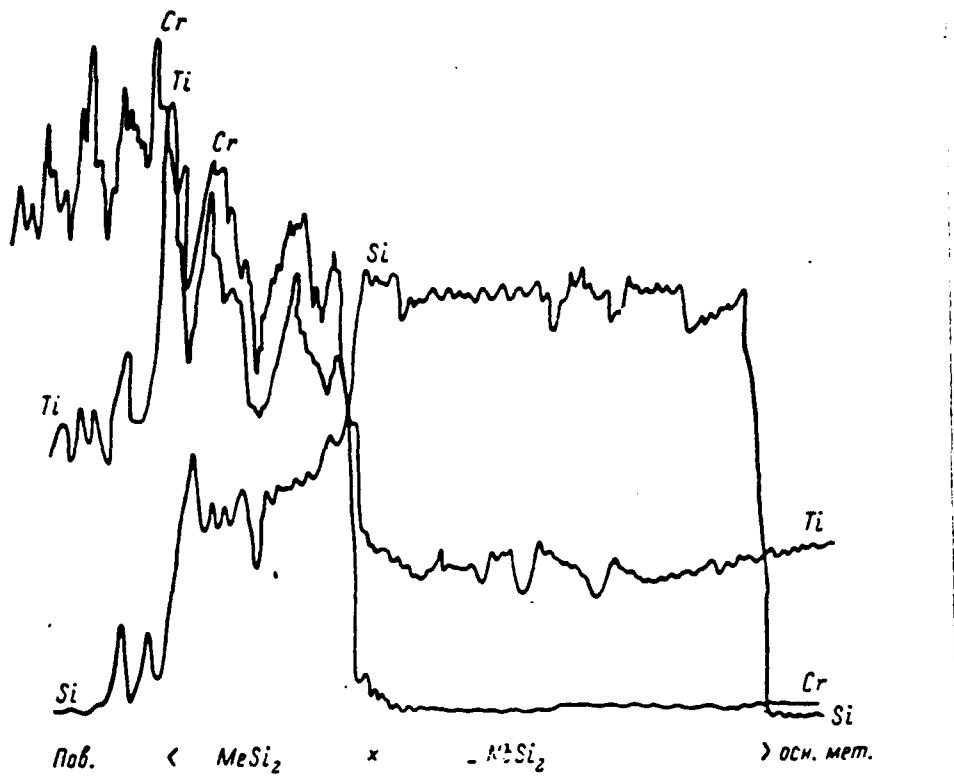


Fig. 6. Distribution of elements in the surface layer of alloy VN-4 with Cr-Ti-Si coating (siliconized in a mixture containing a reduced amount of chlorides).

As a result of diffusion of the silicon in the base metal, the phase Nb_5Si_3 is formed, which is found after prolonged heating on samples with chromium-aluminum-silicon and with chromium-titanium-silicon coatings.

Conclusions

1. The method of by-layer phase analysis and microsound have determined that in chromalitization and in chromotitanization of niobium alloys VN-2A and VN-4 the diffusion layer consists of the phase Cr_2Nb . Upon subsequent siliconization, a layer consisting primarily of the phase $NbSi_2$ is formed.

2. Heating alloys with coatings in air at high temperatures leads to the formation of oxides on the surface: SiO_2 (on coating Cr-Al-Si), Cr_2O_3 , SiO_2 , TiO_2 , $\beta-Nb_2O_5$ (on coating Cr-Ti-Si), Cr_2O_3 (on coating Cr-Ti-Si upon siliconization in a mixture with reduced amount of chlorides). As a result of diffusion of the silicon into the alloy, the phase Nb_5Si_3 is formed between the phase $NbSi_2$ and the base metal.

3. An explanation of the mechanism of forming the diffuse layer in the dual process --chromalitization (chromotitanization) with subsequent siliconization has been proposed.

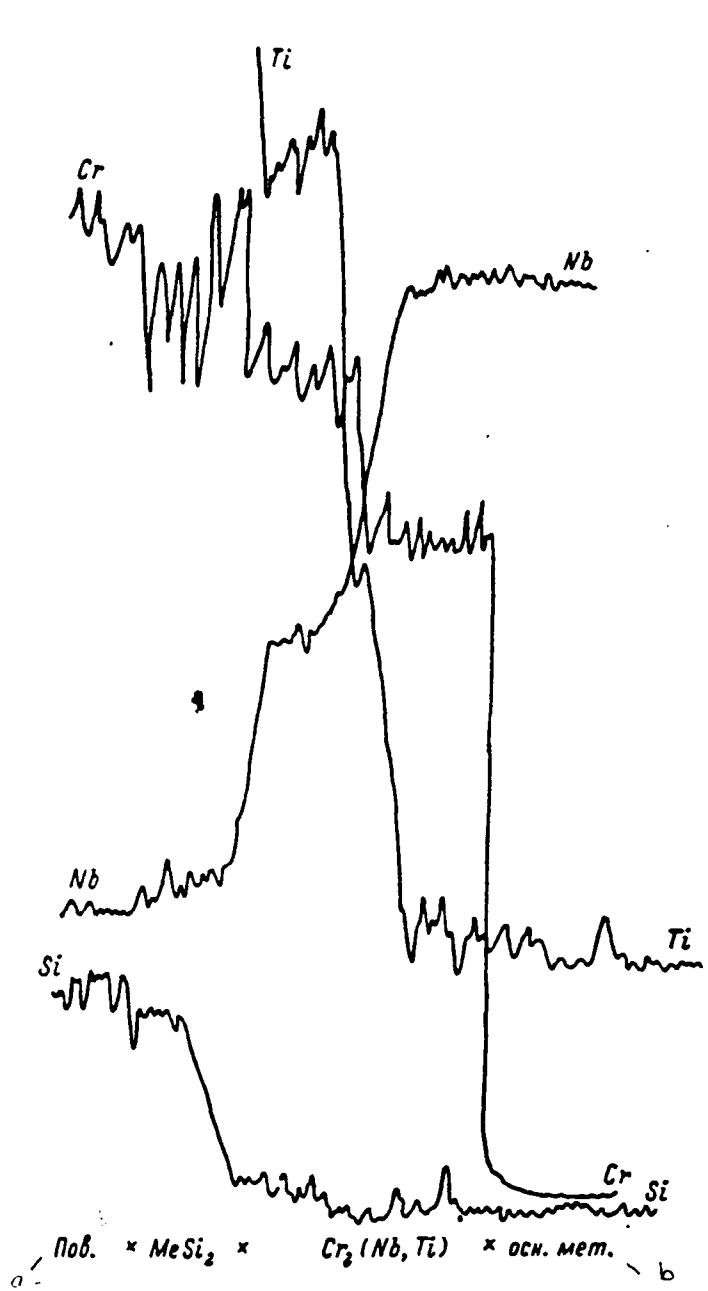


Fig. 7. Distribution of elements in surface layer of alloy VN-4 with coating Cr-Ti-Si (siliconization in mixture without chlorides).
 a - surface; b - base metal.

Literature

1. Ye. L. Zarubina, N. F. Lashko, A. F. Platonova, A. N. Sokolov.
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